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FLAVOR MODIFICATION OF LOW-GRADE HONEY[1]

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Flavor modification of certain strong-flavored types of honey has been discussed for years (1); in fact, several patents disclose methods for "refining" honey (2). There is considerable interest at present in such flavor modification as a means for better utilization of a considerable quantity of dark, unmarketable honey now produced each year in this country.

There are two alternatives to be considered. The first involves the production of a material that would retain most of its desirable honey characteristics, including some measure of honey flavor. The second is production of a completely deflavored honey sirup. Such a material would be in direct competition with invert sirup, and would be suitable for use in substantially all of the present applications of the latter.

We have studied both these types of treatment. Nearly 200 small-scale processing trials have been made, the majority of them concerned with production of a sirup with as much honey character as possible.

Production of Milder-Flavored "Honey"

Lothrop and Paine, who are well known in the honey industry for their method for filtration of full-bodied honey, did considerable research on the colloids of honey. In an article published in 1931 (3), they describe a process for removing a considerable proportion of such material from diluted honey by treatment with bentonite clay. We have used this process substantially as described by them as a means of producing a milder-flavored product from certain honeys. The following process was applied to several strong-flavored honeys, and the results are shown in Table 1.

The honey was diluted to about 50% solids, warmed to 150° F. (66° C.) and the required amount (see test below) of 5% bentonite suspension added. After 20 minutes agitation the mixture was filtered under pressure with 2 to 2-1/2% of a suitable filter aid on a precoated surface, and vacuum-evaporated to honey density.

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[1] Report of a study made under the Research and Marketing Act of 1946.

[2] One of the laboratories of the Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, United States Department of Agriculture.

To determine the amount of bentonite[3] required, the test described by Lothrop and Paine is used. To six 25-ml. portions of a 40% solids honey solution in test tubes are added 0.14, 0.28, 0.58, 1.15, 1.72, and 2.3 ml. of the 5% bentonite suspension. The tubes are then shaken and let stand until coagulation is complete (usually 5 to 20 minutes). The correct amount of bentonite is indicated by the tube in which the supernatant liquid is clear with a minimum volume of precipitated material. The amount of bentonite required for a large batch may be calculated from the amount added to this tube. The number of ml. added when multiplied by 7 gives the number of pounds of 5% bentonite suspension to add per 100 pounds of honey.

The principal difficulty encountered in the process was that of filtering the bentonite-treated honey sirup. It has been found, however, that this difficulty is not caused by the bentonite, but rather by the nature of the material removed from the honey. Many other treatments were examined as alternate to the bentonite treatment but none gave desirable products with easier filtration. Filtration rates shown in Table 1 are too slow for large-scale operation (except that given for horsemint honey). Further dilution would be required for proper filtration.

Bentonite treatment gave products somewhat lighter in color, lower in acidity, and of definitely milder flavor. It will not make a clover honey from a buckwheat honey, however. The treatment did not produce an acceptable product from several smartweed honeys. Though their flavor was improved, the characteristic "weedy" after-taste was not eliminated.

Table 2 shows analyses of three honeys before and after bentonite treatment. The values for the treated honey products have been calculated to the same moisture content as the original honey in each case. Analyses were done by the methods of the Association of Official Agricultural Chemists (4). The most significant change effected by bentonite treatment, the elimination of colloidal material, is reflected in the changes in nitrogen content shown in the table. Paine, Gertler, and Lothrop (5) reported that removal of colloids from honey reduced the nitrogen content to about half of the original value.

After noting that the process was quite beneficial with respect to flavor when applied to buckwheat honey, Lothrop and Paine stated, "This process promises to be valuable as a means of improving the quality and marketability of low-grade honeys." To our knowledge, no attempts have been made in the intervening nineteen years to apply the process on a commercial scale, possibly because it was believed at the time that no process which required dilution of the honey was feasible. At present, however, use of vacuum evaporation equipment by honey processing plants is not uncommon; furthermore there has been a trend to larger-scale cooperative packing and processing which would allow operation of such a process on a larger, more economical scale.

[3] The bentonite must be of the swelling, gel-forming, sodium type. Suitable materials are "Volclay Bentonite 325 mesh', 'BC Volclay', or 'KWK Volclay'. In giving the trade names mentioned here and elsewhere in this publication, the Bureau of Agricultural and Industrial Chemistry United States Department of Agriculture, does not in any way guarantee these products nor are they recommended in preference to similar products not mentioned.

Honey products of the type shown in Tables 1 and 2 still possess honey flavor and should command a premium over the completely deflavored products to be described later. They can be standardized as to moisture content, and acidity; have pronounced but not rank flavors; and have a reduced colloid content. Such reduction in colloid content should have a beneficial effect in several respects. According to Lothrop and Paine (3,6) the colloids of honey are largely responsible for the low caramelization temperature of honey when compared with aqueous solutions of the same sugars. The effect of colloids on color (7), viscosity (8), surface tension (9, turbidity (10), and granulation (11) of honey has been discussed by these authors. The higher colloid content possessed by certain strong-flavored honeys (5) may be a possible source of difficulty when they are used in cooking, baking and confectionery. In other words, we have here a relatively simple process which may be used to "standardize " certain strong flavored honeys for industrial uses where definite flavor is desired. Application of such processing to lighter, mild honeys is not desirable since the colloid content of these honeys is not high enough to cause difficulties in normal use, nor is the flavor objectionable (3).

Whether a bentonite-treated honey could still be labeled "honey", or the word "honey", suitably qualified, used on the label must be determined by the United States Food and Drug Administration if the product is to be shipped in interstate commerce.

Production of a Deflavored Honey Product

A suitable process for deflavoring honey involves (a) dilution of the honey with water sufficiently to permit filtration, (b) addition of lime to neutralize part of the acidity, (c) agitation at an elevated temperature with a suitable activated carbon, (d) filtration with adequate filter aid on a precoated filter press, and (e) vacuum evaporation of the deflavored sirup to the desired density.

1 **Dilution** - All honeys were diluted for treatment to facilitate filtration. The degree of dilution necessary depends on the honey and the equipment available. A minimum filtration rate of about 4 gallons per square foot of filter surface per hour (on the dilute basis) must be realized; even this is slow. Table 3 shows the relation between refractive index of dilute honey solutions, their solids content, and their sucrose equivalent (Brix). It may be used to determine the solids content of crude and carbon-treated honey sirups by refractometer. If the refractometer has a refractive index scale, columns 1 and 3 should be used. If the instrument has only a "percent sucrose" scale such as the hand refractometer, columns 2 and 3 should be used.

2. **Liming** - The acidity of all diluted honeys was adjusted to about pH 4.3 with a slurry of hydrated lime. The reason for this is twofold: the absence of specific flavor in the product makes the natural acidity too pronounced for some purposes, and the stability of the colloidal material is

least at pH values near [illegible], its isoelectric point (3). At the isoelectric point the colloids tend to separate out of the solution, hence they are more easily removed. It will be noted that the various honeys require differing amounts of lime for this acidity adjustment.

The pH should be adjusted to a value between 4.1 and 4.4. Because of the color of the diluted honey, colorimetric methods for determination of pH are not sufficiently sensitive. Best control of this step is obtained by use of an electrically operated pH meter. Because such equipment is expensive, an alternative method of determining the end-point in the liming step is given below.

3. **Heating** - No deleterious effect of heating the mixture to at least 180° F. (82°C.) has been noted. A minimum temperature of about 150° F. (66° C.) is suggested with a minimum time of treatment of about 20 minutes. Treatment to one hour had no further effect on the product.

4. **Carbon** - Certain activated carbons are recommended by their manufacturers for treatment of such solutions. They are Darco S-51, Darco KB, Darco G-60, Nuchar C-190N, and Nuchar C. Certain of these, Darco S-51 and Nuchar C 190-N, are more economical than others.

5. **Filter Aid** - Diatomaceous filter aids are essential in this process. The particle size of the filter aid should be more or less proportional to that of the carbon being used[4]. In these experiments, Dicalite "Special Speedflo" or Johns Manville "Celite #512" were used. Some improvement in filtration rate might be attained by using somewhat coarser filter aids. The amount should be roughly equal to or slightly exceed the weight of carbon to be filtered out of the honey and should be added dry to the batch to be filtered.

6. **Precoating** - The filter press should be precoated by standard procedures, with an amount of filter aid to equal 0.1 pound per square foot of filter area in a volume of hot water one and one-half times the liquid volume held by the filter press, ending with about 5 pounds per square inch pressure.

7. **Filtration** - Filtration is carried out in the usual manner for the filter press used. Care must be taken to keep carbon and filter aid in suspension during the filtration. See below for details of filtration.

8. **Cooling** - Unless the filtrate is run directly into the vacuum pan it should be cooled as it comes from the filter. If large scale operation is carried out this may be done on a heat exchanger, using the hot filtrate to heat the honey sirup for the next batch. Otherwise, the filtrate may be water cooled in a dairy-type cooler.

9 **Evaporation** - The filtrate must be evaporated in a vacuum evaporator at a temperature of about 150° F. (66° C.) maximum to a solids content of 81 to 85%

[4] SPECIFIC ADVICE AS TO SELECTION OF FILTER AID MAY BE OBTAINED FROM THE MANUFACTURER.

Test for lime requirement

The procedure described below requires no expensive equipment and is sufficiently accurate for the purpose of determining the amount of lime necessary to adjust the acidity of a honey to the desired value. It depends on the principle that when the acidity of a dilute honey solution is near the isoelectric point of the colloids, a visible coagulation takes place.

Solutions required.

1. **Saturated lime water at 25° C. (77° F.).** This is the *clear* liquid from a well stoppered suspension of hydrated lime [$Ca(OH)_2$] in water (one ounce lime in one gallon water) which has been well shaken and then allowed to settle. Make fresh weekly.

2. **Lime slurry.** This is a mixture containing one pound seven ounces of dry hydrated lime in one gallon of water. Shake well before using.

Equipment required:

	Approx. Cost
1 - 10 ml. serological pipet	$ 1.50
1 - 100 ml. graduated cylinder	1.20
6 - Culture tubes (test tube) 25 x 150 mm	.60
1 - Thermometer, laboratory -10° +110° C. (or 0 to 220° F.)	1.50
	$ 4.80

Procedure:

Mix well one volume of honey with exactly two volumes of hot water. Place 30 ml. of this in each test tube. With the pipet place in successive numbered tubes 0, 2, 4, 6, 8, 10 ml. of clear limewater, shake. Put the tubes in a pan of hot water with a thermometer in one tube and heat until the thermometer shows 80° C. (176° F.). Immediately remove all tubes. The lowest numbered tube in which coagulation appears is the indicating tube, showing how much lime is to be added to the main batch of honey. For each ml. of limewater in this tube, add one fluid ounce of the well-shaken lime slurry for each 100 pounds of honey in the batch to be treated.

Details of Filtration

Because of the relatively large amount of solids being removed from the honey mixture, considerable loss of yield ("shrinkage") will be encountered by retention in the filter cake unless certain filtration practices are followed. These procedures are designed to minimize losses of sugar in processing. They are: (1) "Sweetening off" of the filter cake when the process has reached capacity. This may be done by pumping hot water through the press, collecting the wash water with the deflavored honey product until a solids content of 20 to 30% is reached, then diverting the filtrate to a make-up tank for the next batch. Another, less efficient way is to take down the press, agitate the cake with water and refilter, using the filtrate for make-up of the next batch or adding to the product, depending on density. (2) In precoating the press, a water-carried precoat should be used, since recirculation of the processing batch for precoating may lead to early blockage of the press. When "sweetening on", or starting the carbon-containing material through the press, the first liquid through the press will be water, increasing in density to the full density of the solution being filtered. Use of this fore-run for make-up on the next batch will also reduce sugar losses.

Results and Discussion

Treatment of honeys by relatively drastic means to eliminate completely any honey flavor and to reduce color considerably is not new. Nearly 20 years ago a packer deflavored about 350,000 pounds of buckwheat honey. Gardner (12) points out that during World War II one processor alone deflavored as much as 70,000 pounds of imported honey daily. Technically, therefore there is nothing new in producing a completely deflavored product. Difficulties encountered are principally economic: (a) Special equipment is required; (b) Cost of treatment must be kept low, and (c) The product must compete with other low-cost flavorless sweetening agents for a market.

The exact procedures to be followed depend somewhat upon the honey type. It has been found, for example, that buckwheat and smartweed honeys are more difficult to filter than fall flower, horsemint, or blends of clover with strong-flavored components, such as buckwheat or heartsease.

Table 4 shows the conditions and materials used to deflavor various honeys in the laboratory. These are all small-scale experiments, each involving about a pound of honey, done under carefully controlled conditions. Filtration was by a small air pressure filter; filtration data below are comparable but probably not equivalent to filtration rates that would be obtained on large-scale practice.

The data recorded in Table 4 are selected from many experiments. All the products of these runs were simply sweet in flavor, having no honey flavor or aftertaste. The amounts of carbon used were about the minimum necessary to remove the flavor completely. The products from buckwheat and fall flower honeys, especially, darkened rapidly in color after processing but did not become as dark as the original honey. The bland flavor was not affected. No advantage was found on treating honey with both bentonite and carbon over carbon treatment alone.

Table 5 shows the composition of four typical deflavored honeys. As in Table 2, the values for the treated honeys have been calculated to the moisture contents of the original honeys. Except for the lowered nitrogen values and slightly higher ash content, the deflavored products are quite similar to the original honeys.

A cost estimation of the process for deflavoring honey as outlined above has been made by Mr. C. S. Redfield of the Chemical Engineering and Development Division of this laboratory. The basis for the estimate was the processing of 18,000 pounds of honey per 8-hour day, producing a 92% yield of deflavored product. It was assumed that plant space, lighting and heating, and ample boiler capacity were available, and a rental charge is made against the process. With $11,870 for equipment, a total capital cost of $26,200, it was estimated that the above process could be carried out at the indicated scale of operations for a production cost of $0.0144 per pound For example, based on seven-cent honey (7.6 cents including the 8% shrinkage) the (total) cost per pound of product would be $0.0904, including amortization of capital investment, but not including administration and sales expense.

Uses of Deflavored Honey

Low-grade, strong honeys after treatment by this process are modified to such an extent that they may no longer be termed "honey", and products containing them should not have "honey" listed as a component. If necessary, a suitable description, subject to Food and Drug approval, such as "refined honey sirup" or "refined honey sugars" or "deflavored honey", might be used.

The deflavored honey product is a perfectly wholesome material, and contains the original honey sugars in substantially the original proportions. The ash content is somewhat higher, nitrogen and colloid content lower Thus, deflavored honeys should answer any purpose served by any other wholesome sweetening agent, in confectionery, beverage, baking or other industries. Their use in such applications would seem primarily to be a matter of cost relative to other sweeteners.

However, a greater return should be achieved if the product could assume a higher value because of special properties not possessed by competitive sweetening agents. A property which these deflavored honeys have by virtue of their production from natural honey is their higher content of levulose than dextrose. One possible use for deflavored honey is as the sugar base in a new crystallized fruit spread. Research at this laboratory has developed such a product (13) made from fruit, fruit juice and deflavored honey sirup, concentrated to honey density and finely crystallized. This is a product in which best results are obtained by using deflavored honey sirup rather than other sweetening materials.

Acknowledgment

We are indebted to the Analytical and Physical Chemistry Division for many of the analyses reported in this paper. We also acknowledge gifts of honey for this work from Sioux Honey Association, Finger Lakes Honey Producers Cooperative, Mr. Vaughn Wilson and Mr. Raymond Fischer.

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Table I

Experimental Data from Laboratory Production of Milder Flavored Honeys

	Bentonite Added	Filtration		Product			
		Dilution[2]	Rate[3]	Solids	Color	pH	Flavor
	%[1]			%	mm[4]		
Buckwheat (a)[5]	---	---	---	81.8	122	3.8	Strong
(b)	0.4	50	2.1	81.6	103	4.1	Mild, Buckwheat
Fall Flowers 1947 (a)	---	---	---	81.5	127	3.7	Strong
(b)	0.4	55	2.6	82.5	117	4.0	Mild
Fall Flowers 1948 (a)	---	---	---	81.9	71	3.8	Somewhat strong
(b)	0.4	65	2.5	82.6	41	3.9	Mild, spicy
Horsemint (a)	---	---	---	81.6	33	3.6	Weedy aftertaste
(b)	0.35	60	9.9	83.9	20	4.1[6]	Spicy, no aftertaste

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[1] *Calculated on original honey basis*

[2] *Percent solids of honey sirup when filtered*

[3] *Gallons per square foot per hour (dilute basis)*

[4] *Millimeters on Pfund color grader*

[5] *(a) Original honey. (b) Processed*

[6] *Limed to pH 4.1 before addition of bentonite*

Table 2

Effect of Bentonite Treatment upon Composition[1] of Honey

	1948 Buckwheat		1948 Fall Flowers		1948 Horsemint	
	Original	Treated[2]	Original	Treated[2]	Original	Treated[2]
Moisture	18.24	18.24	18.20	18.20	18.48	18.48
Total Sugars as invert	74.5	74.8	74.5	74.9	75.9	74.8
Dextrose	36.3	36.5	37.2	34.6	36.0	32.0
Levulose	38.7	37.7	38.2	39.1	39.9	41.3
Sucrose	0.7	1.7	0.3	2.4	1.3	2.9
Ash	0.11	0.18	0.18	0.22	0.15	0.23
Nitrogen	0.16	0.04	0.13	0.07	0.06	0.03
Acidity[3]	42.4	31.8	31.7	18.1	51.2	19.1[4]
Dextrin	0.82	0.49	1.18	0.37	0.71	0.63

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[1] *Expressed as percent of honey at indicated moisture content. Sucrose calculated from difference between reducing sugars before and after acid inversion; levulose determined by low- and high-temperature polarization; dextrose calculated from difference between reducing sugars before inversion and levulose (4).*

[2] *Values calculated to same moisture content as original honey*

[3] *ml .1 N NaOH per 100 gm. honey.*

[4] *Limed to pH 4.1 before addition of bentonite.*

Table 3

Relations Among Refractive Index, Sucrose Equivalents, (Brix) and Total Solids in Diluted Honeys

Ref. Index at 20 °C.	Sucrose Equivalent[1]	Honey Solids%	Ref. Index at 20°C.	Sucrose Equivalent	Honey Solids%
1.4763	74.5	76.0	1.4334	56.2	57.0
1.4740	73.6	75.0	1.4312	55.2	56.0
1.4717	72.7	74.0	1.4291	54.3	55.0
1.4693	71.7	73.0	1.4270	53.3	54.0
1.4670	70.8	72.0	1.4250	52.3	53.0
1.4647	69.8	71.0	1.4230	51.4	52.0
1.4624	68.9	70.0	1.4209	50.4	51.0
1.4600	67.9	69.0	1.4188	49.4	50.0
1.4577	66.9	68.0	1.4167	48.4	49.0
1.4554	65.9	67.0	1.4146	47.4	48.0
1.4531	64.9	66.0	1.4126	46.5	47.0
1.4509	64.0	65.0	1.4106	45.5	46.0
1.4487	63.0	64.0	1.4086	44.5	45.0
1.4465	62.0	63.0	1.4066	43.5	44.0
1.4443	61.1	62.0	1.4047	42.5	43.0
1.4421	60.1	61.0	1.4027	41.5	42.0
1.4399	59.1	60.0	1.4008	40.6	41.0
1.4377	58.2	59.0	1.3988	39.6	40.0
1.4356	57.2	58.0			

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[1] *Rounded to 1 decimal place from table 122, Circular C440, National Bureau of Standards. 1942.*

Table 4

Experimental Data from Laboratory Production of Deflavored Honeys

Pretreatment		Carbon Treatment				Filtration		Reconstituted Product	
Dilution °Bx	Lime %	Kind	% (1)	Temp. °F.	Time min.	Filter Aid % (1)	Filter Rate (2)	pH	Color (3) mm
1947 Buckwheat (Color 140, pH 4.03)									
56	.05	Nuchar C	2.5	145	30	2	4.8	4.18	47
57	.067	Nuc. C190N	2.5	150	30	2.5	5.0	4.32	60
1948 Buckwheat (Color 122, pH 3.81)									
55	.077	Darco KB	1.5	150	30	2.5	9.9	4.25	48
55	.040	Nuc. C190N	1.5	150	30	2.5	8.2	4.30	37
1947 Fall Flowers (Color 127, pH 3.65)									
60	.05	Nuc. C190N	1.5	150	30	2	3.4	4.28	44
55	.054	Nuc. C190N	1.5	150	30	2	7.2	4.27	44
1948 Fall Flowers (Color 71, pH 3.84)									
65	.035	Nuc. C190N	1	150	30	2	15.3	4.30	29
65	.035	Nuc. C190N	1	150	60	2	15.5	4.35	27
65	.035	Darco G-60	1	150	30	2	33.6	4.18	46
1948 Clover-Heartsease (Color 50, pH 3.82)									
65	.018	Nuc. C190N	0.75	150	30	1	24.0	4.38	12
1947 Smartweed (Color 118, pH 4.26)									
55	0.0	Nuc. C190N	3	150	30	2.5	4.9	4.42	8
55	0.0	Darco S-51	3	150	30	2.5	2.6	4.37	18
55	0.0	Nuc. C	3	150	30	2.5	4.2	4.26	5
1948 Smartweed (Color 72, pH 4.25)									
60	0.0	Nuc. C190N	2	150	30	2	4.1	4.35	3
60	0.0	Nuc. C	2	150	30	2	4.5	4.35	7
60	0.0	Darco S51	3	150	30	2.5	6.8	4.45	7
1948 Horsemint (Color 33, pH 3.55)									
65	.08	Nuc. C190N	1	150	30	2	11.9	4.15	4
65	.08	Darco S51	1.5	150	30	2	20.0	4.15	0

(1) Calc. on original honey basis.
(2) In gal. dilute sirup per square ft. per hour.
(3) Color in mm on Pfund honey grader.

Table 5

Effect of Carbon Treatment upon Composition[1] of Honey

	1948 Buckwheat		1948 Fall Flower		1948 Smartweed		1948 Horsemint	
	Orig.	Treated[2]	Orig.	Treated[2]	Orig.	Treated[2]	Orig.	Treated[2]
Moisture	18.24	18.24	18.20	18.20	17.60	17.60	18.48	18.48
Total sugars as invert	74.5	73.8	74.5	74.1	73.3	73.0	75.9	74.6
Dextrose	36.3	36.4	37.2	33.8	31.2	29.0	36.0	32.7
Levulose	38.7	38.3	38.2	40.2	42.2	43.5	39.9	40.5
Sucrose	0.7	0.2	0.26	1.35	2.0	2.1	1.3	2.7
Ash	0.11	0.20	0.18	0.19	0.17	0.20	0.15	0.25
Nitrogen	0.16	0.07	0.13	0.07	0.16	0.06	0.06	0.04
Acidity[3]	42.4	27.5	31.7	20.2	32.4	16.3	51.2	17.1
Dextrin	0.82	0.76	1.18	0.88	1.14	0.98	0.71	0.96

[1] *Expressed as percent of honey at indicated moisture content. Sucrose calculated from difference between reducing sugars before and after acid inversion; levulose determined by low- and high- temperature polarization; dextrose calculated from difference between reducing sugars before inversion and levulose (4).*

[2] *Values calculated to same moisture content as original honey.*

[3] *ml 0.1 N NaOH per 100 gm. honey.*